

ATTACHMENT B

ENSR DATA VALIDATION PROTOCOLS

Full: _____
 Limited: _____

ENSR Data Pkg#: _____
 Site Name: _____
 Project Number: _____

REVIEW OF DIOXIN/FURAN DATA PACKAGE

The following guidelines for evaluating dioxins and/or furans were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decisions and in better serving the needs of the data users. Quality control validation criteria were derived from United States Environmental Protection Agency (USEPA) publications: *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846* (Final Update III, December 1996), specifically SW-846 Method 8290 *Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)*. Validation actions were derived from *USEPA Analytical Operations/Data Quality Center (AOC), National Functional Guidelines For Chlorinated Dioxin/Furan Data Review*, (Final August 2002).

The hardcopied (laboratory name) _____ data package has been reviewed and the quality assurance and performance data summarized.

The data review for dioxins/furans included the following samples:

Lab Project No.	_____	Sampling Date(s)	_____
No. of Samples	_____	Shipping Date(s)	_____
Sample Matrix	_____	Date(s) Rec'd by Lab	_____

Equipment Blank IDs: _____

 Field Blank IDs: _____

 Field Duplicate IDs: _____

The general criteria used to determine the performance were based on an examination of the following:

___ Data Completeness	___ Laboratory Control Sample
___ Holding Times	___ Field Duplicates
___ GC/MS Performance Checks	___ Internal Standard Recoveries
___ Calibrations	___ Compound Identification
___ Blanks	___ Compound Quantification
___ Matrix Spike/Matrix Spike Duplicate	___ Percent Solids

Overall Comments: _____

Reviewer: _____ Date: _____

NATIONAL FUNCTIONAL GUIDELINES DIOXIN/FURAN DATA QUALIFIER DEFINITIONS

- U - The analyte was analyzed for but not detected.
- J - The analyte was positively identified. The associated numerical value is the approximate concentration of the analyte in the sample.
- N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".
- NJ - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
- UJ - The analyte was not detected. However, the reported detection limit is approximated and may be inaccurate or imprecise.
- R - The sample results are unusable. The analyte may or may not be present in the sample.

I. DATA COMPLETENESS

A. Data Package:

Missing Information

Date Lab ContactedDate Received[illegible]

B. Other Discrepancies:

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II. HOLDING TIMES

The objective of this parameter is to ascertain the validity of results based on the holding time of the sample from the time of collection to the time of extraction, and subsequently from the time of extraction to the time of analysis.

Complete table for all samples and circle the extraction and/or analysis date for samples not within criteria.

Sample ID	Date Sampled	Date Extracted	Date Analyzed	Action

Cooler Temperature(s): _____

Preservation Criteria:

Waters - If residual chlorine is present, treat the sample with sodium thiosulfate and if pH >9, adjust the pH to between 7-9 with sulfuric acid. Cooler temperatures must be at $2-4^{\circ}\text{C} \pm 2^{\circ}$. Samples must be stored in the dark

Soils - Cooler temperatures must be $2-4^{\circ}\text{C} \pm 2^{\circ}$. Samples must be stored in the dark.

Tissue - Samples must be stored at -20°C . Samples must be stored in the dark.

Holding Time Criteria for Waters, Soils, and Tissues:

Extract within 30 days of sample collection and analyze within 45 days of extraction

Actions:

If the samples were not properly preserved, qualify as estimated (J and UJ) all positive and non-detect results.

If holding times are exceeded, qualify as estimated (J and UJ) all positive and non-detect results.

If holding times are grossly exceeded, (>60 days for extraction and >90 days for analysis), qualify as estimates positive results (J) and reject (R) the non-detect results.

III. GC/MS TUNING & PERFORMANCE CHECK

GC/MS instrument performance checks are performed to ensure proper mass resolution, identification, and sensitivity.

A. MS Resolution – Perfluorokerosene (PFK) Molecular Leak:

1. Was the PFK molecular leak performed at the proper frequency? Yes No

Criteria: Beginning and end of each 12-hour period of operation

2. If calculated resolution results **are** available, was the resolution greater than or equal to 10,000 (10% valley) at m/z 304.9824 (PFK) or any reference signal close to m/z 303.9016 (TCDF)?

Yes No Unavailable

3. For each descriptor listed in Table 6 were reference peaks selected that cover the mass range of the descriptor? Yes No

4. If calculated results **are** available for each descriptor, was the resolution greater than or equal to 10,000 (10% valley) and the deviation between the exact m/z and the theoretical m/z (in Table 6) for each exact m/z less than 5 ppm? Yes No Unavailable

5. If calculated results for resolution and deviation from exact m/z **are not** available for each descriptor, visually inspect the shape of the peak profiles for symmetry and baseline. Were the peak shapes symmetrical and were the baselines adequate? Yes No

Actions:

If the mass spectrometer resolution is <10,000, all of the associated data should be rejected (R) If no for any of the other above questions, use professional judgment in the qualification of the data. Explain actions and list affected samples below.

B. GC Column Performance Check – Window Defining Mixture (WDM)

1. Was the GC column performance check (*i.e.* WDM) performed at the proper frequency? Yes No

Criteria: Beginning of each 12-hour period during which samples are analyzed and prior to initial calibration.

2. Are the 1st and last isomers in each homologue series present in the WDM? Yes No

Actions:

If no to either 1 or 2, but the calibration standards met specifications, then the individual 2,3,7,8-substituted congener results may be usable without qualification. Total homologue results, however, should be qualified as estimated (J and UJ) since one or more CDDs/CDFs may not have been detected. If the calibration standards indicate a significant problem with the descriptor switching times, all the associated results should be qualified as rejected (R).

III. GC/MS PERFORMANCE CHECKS (continued)

3. Was the chromatographic separation between 2,3,7,8-TCDD and the peaks representing other unlabeled TCDD isomers resolved with a valley of $\leq 25\%$? Yes No

List performance checks which did not meet this resolution criteria and the associated samples below.

Check Standard ID	Valley %	Associated Samples

Actions:

If the GC resolution does not meet the criteria, qualify as estimated (J and UJ) the positive results and non-detect results for tetras, pentas, and hexas (dioxins and furans). The hepta isomers are not believed to be affected. OCDD and OCDF are not affected as there is only one isomer in each group. Non-detects are not affected.

4. Were the absolute retention times for the switching of SIM ions from one homologous series to the next higher homologous series greater than 10 seconds apart? Yes No

Note: Be sure to check for adequate separation between 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF since these elute within 15 seconds of each other.

Action:

If the switching times are less than 10 seconds apart, this may result in false negative or low biased results for some of the congeners or totals. Use professional judgment and qualify as estimated (J and UJ) all positive and non-detect results with retention time shifts greater than 10 seconds of the corresponding homologue.

IV. CALIBRATIONS

A. Initial Calibration

1. Were the five concentration calibration solutions listed in Table 5 of the method utilized in the initial calibration of the instrument (particularly the lowest calibration standard)?

Yes No

Action: If no, use professional judgment in the qualification of the data.

2. Were SIM data acquired for each of the ions listed in Table 6 of the method? Yes No

Action:

If no, ask lab for an explanation. If an incorrect ion was used, reject (R) all associated data for the affected analyte.

3. Retention Times (RTs)

- a. For 2,3,7,8-substituted congeners which have an isotopically labeled internal or recovery standard, the RT of the 2 ions must be within -1 to +3 seconds of the isotopically labeled standard.
- b. For 2,3,7,8-substituted congeners which do not have an isotopically labeled internal or recovery standard, the RT of the 2 ions must fall within 0.005 RT units of the Relative RT (RRT) measured in the calibration standard. (Note: Identification of OCDF is based on its RT relative to ¹³C₁₂-OCDD).

Action:

If the above criteria are not met, qualify all associated results as rejected (R).

4. The ion abundance ratios for all compounds in all standards must be evaluated. List the ion abundance ratios which are outside the acceptance criteria.

Criteria: Table 8 of the method lists the ion abundance ratio acceptance criteria.

Standard ID	Ion Ratio	Analyte	Samples Affected

Actions:

If the ion abundance ratio is not met for a 2,3,7,8-substituted congener (see Table below for limits), qualify as rejected (R) all associated sample results for compounds with failed ion ratios in the initial calibration. At the reviewer's discretion, a more in-depth review to minimize the amount of data rejected may be accomplished by the following:

- If the ion abundance ratio is outside the limits for an analyte in the HRCC-1 solution, then low-end results for that analyte (below the HRCC-2 standard) should be qualified as rejected (R).
- If the ion abundance ratio is outside the limits for an analyte in the HRCC-5 solution, then high-end results for that analyte (above the HRCC-4 standard) should be qualified as rejected (R).

IV. CALIBRATIONS (continued)

Number of Chlorine Atoms	M/Z's Forming Ratio	Theoretical Ratio	±15 %QC Limits
4 ¹	M/(M+2)	0.77	0.65-0.89
5	(M+2)/M+4)	1.55	1.32-1.78
6	(M+2)/M+4)	1.24	1.05-1.43
6 ²	M/(M+2)	0.51	0.43-0.59
7	(M+2)/M+4)	1.04	0.88-1.20
7 ³	M/(M+2)	0.44	0.37-0.51
8	(M+2)/M+4)	0.89	0.76-1.02

¹-Does not apply to ³⁷Cl₄-2,3,7,8-TCDD (cleanup standard).

²-Used for ¹³Cl₁₂-HxCDF only

³-Used for ¹³Cl₁₂-HpCDF only

5. Were the signal/noise ratios for all peaks greater than or equal to 10?

Yes No

Action:

If no, and if the signal/noise ratio is <10 for any 2,3,7,8-substituted congeners (unlabeled), the instrument sensitivity may be impacted. In this case, all non-detect results in samples associated with this initial calibration should be rejected (R) and the positive results should be estimated (J).

If the signal/noise ratio for a labeled internal standard or recovery standard are <10, sensitivity of the instrument may have been impacted or the standard was not properly spiked. Use professional judgment to determine effect on data quality.

6. List the percent relative standard deviations (%RSDs) that were outside the QC acceptance criteria of ≤ 20% for all 2,3,7,8-substituted congeners (unlabeled) and ≤ 30% for internal standards (labeled).

7.

DATE	LAB FILE ID#	%RSD	ANALYTE	SAMPLES AFFECTED

Actions:

If the %RSD is >20 for a 2,3,7,8-substituted congener (unlabeled), qualify as estimated (J and UJ) the positive and non-detect results for the affected analyte in samples associated with this initial calibration. If the RSD is ≥ 20% for an unlabeled congener, examine the possibility of directing the RSD to within 20% by discarding either the HRCC-1 or HRCC-5 standard response factors. If discarding either of those two points brings the RSD within 20%, qualify as estimated (J and UJ) the positive and non-detect results associated with the offending portion of the initial calibration (low or high). If non-linearity impacted a majority of data, all positive and non-detect results should be qualified as estimated (J and UJ). Use professional judgment to qualify the data in cases where the internal standard %RSD is ≥ 30%.

IV. CALIBRATIONS (continued)

B. Continuing Calibration

1. Was the calibration verification standard analyzed at the proper frequency (*i.e.* HRCC-3)?
 Yes No

Criteria: Beginning of each 12-hour period of operation and at the end of the 12-hour shift.

Action: If no, use professional judgment in the qualification of the data.

8. Were SIM data acquired for each of the ions listed in Table 6 of the method? Yes No

Action:

If no, ask lab for an explanation. If an incorrect ion was used, qualify as rejected (R) all associated data for the affected analyte.

3. Retention Times

- a. For 2,3,7,8-substituted congeners which have an isotopically labeled internal or recovery standard, the RT of the 2 ions must be within -1 to +3 seconds of the isotopically labeled standard.
- a. For 2,3,7,8-substituted congeners which do not have an isotopically labeled internal or recovery standard, the RT of the 2 ions must fall within 0.005 RT units of the RRT measured in the calibration standard. Note: The identification of OCDF is based on its RT relative to ¹³C₁₂-OCDD.

Actions:

If the retention times for any 2,3,7,8-substituted congeners (unlabeled) in the continuing calibration standard are not within the retention time windows, use professional judgment and qualify as estimated (J and UJ) all positive and non-detect results for the affected analyte in samples associated with this continuing calibration.

If the recovery standard retention times drift by more than ±15 seconds from the initial HRCC-3 analysis and the continuing calibration standard, use professional judgment to qualify all associated sample results. All positive and non-detect results should be rejected (R) unless based on a review of the selected ion current profile (SICP), there appears to be no affect on the results.

4. Do the 2 SIM ions maximize simultaneously (±2 seconds) for each analyte? Yes No

Actions:

Use professional judgment if the required retention times are not met for the 2 SIM ions.

IV. CALIBRATIONS (continued)

5. The ion abundance ratios for all compounds in all standards must be evaluated. List the ion abundance ratios which are outside the acceptance criteria.

Criteria: Table 8 of the method lists the ion abundance ratio acceptance criteria.

Standard ID	Ion Ratio	Affected Compound	Associated Samples

Actions:

If the ion abundance ratio is not met for a 2,3,7,8-substituted congener (see Table below for limits), qualify as estimated (J and UJ) all associated positive and non-detect sample results for compounds with failed ion ratios in the continuing calibration.

Number of Chlorine Atoms	M/Z's Forming Ratio	Theoretical Ratio	±15 %QC Limits
4 ¹	M/(M+2)	0.77	0.65-0.89
5	(M+2)/M+4)	1.55	1.32-1.78
6	(M+2)/M+4)	1.24	1.05-1.43
6 ²	M/(M+2)	0.51	0.43-0.59
7	(M+2)/M+4)	1.04	0.88-1.20
7 ³	M/(M+2)	0.44	0.37-0.51
8	(M+2)/M+4)	0.89	0.76-1.02

¹-Does not apply to ³⁷Cl₄-2,3,7,8-TCDD (cleanup standard).

²-Used for ¹³Cl₁₂-HxCDF only

³-Used for ¹³Cl₁₂-HpCDF only

6. Were the signal/noise ratios for all peaks greater than or equal to 10?

Yes No

Action:

If no, and if the signal/noise ratio is <10 for any 2,3,7,8-substituted congeners (unlabeled), the instrument sensitivity may be impacted. In this case, all non-detect results in samples associated with this continuing calibration should be rejected (R) and the positive results should be estimated (J).

If the signal/noise ratio for a labeled internal standard or recovery standard are <10, sensitivity of the instrument may have been impacted or the standard was not properly spiked. Use professional judgment to determine effect on data quality.

IV. CALIBRATIONS (continued)

7. List the percent difference (%Ds) that were outside the QC acceptance criteria of $\leq 20\%$ for all 2,3,7,8-substituted congeners (unlabeled) and $\leq 30\%$ for internal standards (ending calibration standard QC acceptance criteria of $\leq 25\%$ for all 2,3,7,8-substituted congeners (unlabeled) and $\leq 35\%$ for internal standards).

DATE	LAB FILE ID#	%D	ANALYTE	SAMPLES AFFECTED

Note: for ending calibration standard only: If $\%D > 25\%$ (for any unlabeled compounds) and/or $\%D > 35\%$ (for labeled compounds), verify that the laboratory used the mean response factor (RF) from the beginning and ending continuing calibration standards in sample calculations instead of the mean RF from the initial calibration.

Actions:

Qualify positive results and non-detects as estimated (J and UJ) if the continuing calibration acceptance criterion is exceeded.

V. BLANKS

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply to any blank associated with the samples, including field, equipment and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. Medium and low level blanks must be treated separately.

1. Laboratory Blanks

Date Analyzed	Lab ID	Level/ Matrix	Compound	Concentration/ Unit
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

2. Field and Equipment Blanks

Date Analyzed	Lab ID	Level/ Matrix	Compound	Concentration/ Unit
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

V. BLANKS (continued)

1. Was a method blank extracted with each batch of 20 samples/matrix? Yes No
2. Was the method blank analyzed between the calibration standard and the first sample? Yes No

If no, use professional judgment and explain actions below. If contamination is suspected, use professional judgment to qualify as estimated (J) all positive results associated with the suspected contaminant.

VI. MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long-term precision and accuracy of the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples.

1. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Recoveries and Precision

Level / Matrix: _____

Sample ID: _____

List the percent recoveries (%R) and relative percent differences (RPDs) of spiked analytes which do not meet criteria. Refer to the QAPP for QC acceptance limits. If limits are not listed, apply professional judgment criteria of 40-135%R and 25%RPD.

MS or MSD	Compound	%R or RPD	QC Limits	Action

Actions:

	%R is > Upper QC Limit	%R is ≥ 10% but < Lower QC Limit	%R is < 10%	RPD Outside QC Criteria
Positive results	J	J	J	J
Non-detect results	Accept	UJ	R	UJ

Notes: (1) Qualifications should be applied to the affected compound in the unspiked sample only.

(2) If the majority of spike compound %Rs or RPDs are outside the QC acceptance criteria, use professional judgment to J, UJ, and/or R all compounds in the unspiked sample.

(3) No action is necessary if the concentration in the unspiked sample exceeds 4x the concentration of the spike added.

A separate worksheet should be used for each MS/MSD pair.

VIB. MATRIX SPIKE/MATRIX SPIKE DUPLICATE

2. MS/MSD - Unspiked Compounds

Level / Matrix: _____

Sample ID: _____

List the concentrations of the unspiked compounds and determine the %RSDs of these compounds in the unspiked sample, matrix spike, and matrix spike duplicate.

Compound	Concentration			%RSD	Action
	Sample	MS	MSD		
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

Criteria:

% RSD \leq 50.

Actions:

If the %RSD > 50, qualify the positive or non-detect result in the unspiked sample as estimated (J and UJ).

If the %RSD is not calculable (NC) due to a non-detect value in the sample, MS, and/or MSD, use professional judgment to qualify sample data.

VII. LABORATORY CONTROL SAMPLES

This data is generated to determine accuracy of the analytical method for various matrices.

1. Laboratory Control Sample (LCS) Recoveries

List the percent recoveries (%R) of spiked analytes which do not meet criteria. Refer to the QAPP for QC acceptance limits. If limits are not listed, use professional judgment criteria of 40-135%R.

LCS ID	Compound	%R	QC Limits	Action

Actions:

	%R is > Upper QC Limit	%R is ≥ 10% but < Lower QC Limit	%R is < 10%
Positive results	J	J	J
Non-detect results	Accept	UJ	R

Note: (1) If the LCS exhibits many %Rs which are outside the QC acceptance criteria and this appears to be an isolated, explainable incident affecting the LCS only, use professional judgment in the qualification of sample data.

(2) If the majority of spike compound %Rs are outside the QC acceptance criteria, and there is no reasonable explanation for all the exceedances, use professional judgment to J, UJ, and/or R all compounds in the associated samples.

2. LCS Frequency

1. Was an LCS extracted with each batch of 20 samples/matrix? Yes No

If no, use professional judgment and explain actions below.

VIII. FIELD DUPLICATES

Sample IDs. _____

Matrix: _____

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which measure only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

Criteria:

Soils RPD ≤ 50 , Aqueous RPD ≤ 30 , if both the sample and duplicate results are $\geq 5x$ sample quantitation limit (SQL). The RPD criterion is doubled, if both the sample and duplicate results are $< 5x$ SQL.

Compound	Sample Quantitation Limit	Sample Concentration	Duplicate Sample Concentration	% RPD	Action

Actions:

- If the concentrations in the sample and field duplicate are positive and the RPD criterion is exceeded, qualify the positive results as estimated (J).
- If a dioxin/furan in a field duplicate pair is detected at $\geq 5x$ SQL in one sample and non-detect in the other sample qualify the positive and non-detect results as estimated (J and UJ).
- If a dioxin/furan in a field duplicate pair is non-detect in one sample but detected at $< 5x$ SQL in the other, use professional judgment to qualify sample results.
- If one dioxin/furan is non-detect in a field duplicate pair and in the other sample is $\geq 5x$ SQL; and the SQLs for the sample and duplicate are significantly different, use professional judgment to qualify sample data.

IX. INTERNAL STANDARD RECOVERIES

Isotopically labeled internal standards (IS) (for PCDDs and PCDFs) are added to each sample, LCS, and method blank prior to extraction. These labeled ISs serve as a measure of the extraction efficiency of each sample, LCS, and blank. These compounds are also used for the quantitation of the PCDD and PCDF isomers.

1. Were samples spiked with all internal standards as specified in Table 2 of the method? Yes No
2. List the internal standards and the associated samples that fell outside the QC acceptance criteria of 40-135 %R.

[illegible]

Actions:

If the IS %Rs fall outside the QC acceptance limits, qualify positive and non-detects results (including EMPCs and EDLs) as follows:

	%R is > 135%R	%R is ≥ 10% but < 40%R	%R is < 10%
Positive results	J	J	J
Non-detect results	Accept	UJ	R

Note: (1) Actions are only applicable to the results associated with the failed internal standard.

- (2) If the IS recoveries are low, but the clean-up standard recovery is not, then the recovery problems may be associated with the extraction procedures or related to a particularly difficult matrix.
- (3) If the IS recoveries are low, but the clean-up standard recovery is not, then the recovery problems may be associated with the extraction procedures or related to a particularly difficult matrix.

X. COMPOUND IDENTIFICATION

1. Were SIM data acquired for each of the ions listed in Table 2 of the method? Yes No

Action: If no, ask lab for an explanation. If an incorrect ion was used, reject (R) all associated data for the affected analyte.

2. Retention Times (RTs)

- For 2,3,7,8-substituted congeners which have an isotopically labeled internal or recovery standard, the RT of the 2 ions must be within -1 to +3 seconds of the isotopically labeled standard.
- For 2,3,7,8-substituted congeners which do not have an isotopically labeled internal or recovery standard, the RT of the 2 ions must fall within 0.005 RT units of the RRT measured in the calibration standard. Note: The identification of OCDF is based on its RT relative to ¹³C₁₂-OCDD.
- For non-2,3,7,8-substituted compounds, the RT must be within the homologous RT windows established by analyzing the GC column performance check (*i.e.* WDM).

Actions:

If the retention times for any 2,3,7,8-substituted congeners (labeled and unlabeled) are not within the established retention time windows, the results cannot be positively identified as dioxins/furans and qualify the results as rejected (R).

3. Do the 2 SIM ions maximize simultaneously (±2 seconds) for each analyte? Yes No

Actions:

If the required retention times are not met for the 2 SIM ions, qualify the results as rejected (R).

4. The ion ratios for all compounds in all standards must be evaluated. List the ion ratios which are outside the acceptance criteria.

Criteria: Table 8 of the method lists the ion abundance ratio acceptance criteria.

Standard ID	Ion Ratio	Affected Compound	Associated Samples

X. COMPOUND IDENTIFICATION (continued)

Actions:

National Function Guidelines states that "If ion abundance criteria are not satisfied, then the data should be rejected (R)". However it also states that "professional judgment should always be used in determining the proper identification of analytes". Use the following professional judgment to qualify the data since the method allows for analytes that do not meet ion abundance ratios to be reported as EMPCs:

If the ion abundance ratio is >15% (see Table below) for a 2,3,7,8-substituted congener (unlabeled), but all other identification criteria (signal/noise and retention times) qualify as estimated (J) the positive result in the sample. Confirm that the value is reported as an EMPC by the laboratory. The presence of EMPC should be noted in the validation report narrative

If the ion abundance ratio is outside the limits for an internal standard or recovery standard, the stability of the mass spectra is in question since the analyte cannot be positively identified in the standard. Qualify positive results as estimated (J) and reject (R) the non-detect results.

Number of Chlorine Atoms	M/Z's Forming Ratio	Theoretical Ratio	±15 %QC Limits
4 ¹	M/(M+2)	0.77	0.65-0.89
5	(M+2)/M+4)	1.55	1.32-1.78
6	(M+2)/M+4)	1.24	1.05-1.43
6 ²	M/(M+2)	0.51	0.43-0.59
7	(M+2)/M+4)	1.04	0.88-1.20
7 ³	M/(M+2)	0.44	0.37-0.51
8	(M+2)/M+4)	0.89	0.76-1.02

¹-Does not apply to ³⁷Cl₄-2,3,7,8-TCDD (cleanup standard).

²-Used for ¹³Cl₁₂-HxCDF only

³-Used for ¹³Cl₁₂-HpCDF only

5. Were the signal/noise ratios for all peaks greater than or equal to 2.5 for 2,3,7,8 substituted congeners and >10 for internal standards? Yes No

Actions:

If the signal/noise ratio is <2.5 for 2,3,7,8-substituted congeners (unlabeled) or <10 for internal standards, positive results should be considered to estimated (J).

6. For peaks that were identified as furans, does a signal/noise ratio ≥ 2.5 at the same time in the corresponding polychlorinated diphenyl ether (PCDPE) channel exist and is the retention time relative to the furan isomer within ±2 seconds? Yes No

Actions:

If PCDPE interferences exist, qualify the positive furan result as estimated (J).

If the laboratory did not monitor for PCDPEs, qualify all positive furan data as estimated (J).

X. COMPOUND IDENTIFICATION (continued)

7. Second Column Confirmation

- | | | | |
|----|--|-----|----|
| a. | Was the sample extract re-analyzed on a 30 m DB-225, fused silica capillary column for 2,3,7,8-TCDF? | Yes | No |
| b. | Did the second column meet calibration specifications? | Yes | No |
| c. | Did the laboratory report the concentration of 2,3,7,8-TCDF from the secondary column | Yes | No |

Actions:

National Function Guidelines states that "If ion abundance criteria are not satisfied, then the data should be rejected (R)". However, use the following professional judgment:

If no, the result for 2,3,7,8-TCDF should be reported from the secondary column; the primary column should only be used for confirmation purposes. If 2,3,7,8-TCDF was not confirmed and reported from a DB-5 column, use professional judgment and qualify the result as estimated (J) due to potential lack of specificity.

XI. COMPOUND QUANTITATION

1. Were all analyte concentrations within the instrument calibration range? Yes No

If no, were appropriate dilutions performed? Yes No

If no, list associated samples and effect on sample data.

2. Were estimated detection limits (EDLs) calculated for all 2,3,7,8-substituted isomers that were not identified as positive values? Yes No

3. Example Calculation

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

XII PERCENT SOLIDS

List samples that have $\leq 30\%$ solids:

Actions:

If a soil sample has $>10\%$ solids but $\leq 30\%$ solids, qualify positive results as estimated (J) and reject (R) non-detect results.

If a soil sample has $<10\%$ solids, reject (R) both positive and non-detect results.

Professional judgment may be applied if the laboratory used increased sample weights prior to extraction.

DATA REVIEW WORKSHEETS

Type of Validation Full: _____
Limited: _____ENSR Data Pkg#: _____
Site Name: _____
Project Number: _____

REVIEW OF SEMIVOLATILE ORGANIC DATA PACKAGE

The following guidelines for evaluating semivolatile organics data were created to delineate required validation actions. This document will assist the reviewer in using professional judgement to make more informed decisions and in better serving the needs of the data users. Quality control validation criteria were derived from the USEPA publications *Test Methods for Evaluating Solid Waste, Physical / Chemical Methods SW846* (Final Update III, December 1996), specifically SW-846 methods 8000B/8270C, and the project Quality Assurance Project Plan (QAPP). Validation actions were derived from *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (October 1999) and *Region 5 Standard Operating Procedure for Validation of CLP Organic Data* (USEPA Region 5 Superfund Technical Support Section, February 1997).

The hardcopy data package has been reviewed and the quality assurance and performance data summarized. The data review for semivolatile analytes included:

Laboratory Name/Location _____
Laboratory SDG No. _____
No. of Samples _____
Sample Matrix _____
Equipment/Field Blank ID _____
Field Duplicate IDs _____

The general criteria used to determine the performance were based on an examination of:

___ Data Completeness	___ Matrix Spike/Matrix Spike Duplicate (MS/MSD)
___ Holding Times / Sample Preservation	___ Laboratory Control Samples (LCS)
___ GC/MS Tuning	___ Field Duplicate Precision
___ Calibrations	___ Internal Standard Performance
___ Blank Analysis Results	___ Compound Identification
___ Surrogate Spike Recoveries	___ Quantitation Limits and Sample Results

Overall Comments: _____

Qualifiers:

J - Estimated result.
R - Reject data due to quality control criteria.
U - Compound not detected.
UJ - Estimated nondetect
N - Tentatively identified
JN - Estimated concentration, tentatively identified

Reviewer: _____ Date: _____

DATA REVIEW WORKSHEETS**I. DATA COMPLETENESS****A. Data Package:**

_____ The tests requested on the COC or in subsequent communications were performed and reported

_____ The correct analyte list was reported

_____ The COCs (external and internal) are present and complete

_____ Sample receiving documentation is complete

Missing Information

A. Other Discrepancies:

The objective of this parameter is to ascertain the validity of results based on the holding time (HT) of the sample from the time of collection to the time of extraction, and subsequently from the time of extraction to the time of analysis.

[illegible]

- Extraction HT: Aqueous: Extract within 7 days from sample collection, Soil: Extract within 14 days.
- Analysis HT: Aqueous and Soil: Analysis within 40 days from date of sample extraction.
- Cooler Temperature ($4^{\circ}\text{C} \pm 2^{\circ}\text{C}$): _____

Extraction from sampling (Days)		Analysis from Extraction (Days)	Action Detect/Nondetect
Water	Soil		
1-7	1-14	1-40	Accept
8-14	15-28	--	J/UJ
>14	>28	>40	J/R

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III. GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within standard tuning QC limits.

_____ The DFTPP performance results were reviewed and found to be within the specified criteria of the method. If ion abundance criteria were not met, use professional judgment to qualify results. If mass assignment is in error (e.g., m/z 199 as base peak instead of m/z 198), all associated data are rejected (R).

_____ All samples and CCVs were analyzed within 12 hrs of the DFTPP tunes. If no, use professional judgement to determine if qualification is appropriate.

DATA REVIEW WORKSHEETS

All criteria were met _____
 Criteria were not met
 and/or see below _____

I. CALIBRATIONS

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Dates of Initial Calibration: _____
 Dates of Continuing Calibrations: _____
 Instrument ID Numbers: _____

DATE	Lab File ID#	Analyte	RFs, %RSD, %D, r	Samples Affected

ICAL	Criteria	Action (Detects/Nondetects)
RF	RF \geq 0.05 for all target analytes, including SPCCs	J / R Note: Sample results negated (U) on the basis of blank contamination are not rejected, but estimated (UJ).
%RSD or correlation coefficient (r)	%RSD \leq 30% for CCCs %RSD \leq 15% for other analytes If %RSD > 15, line or curve must have $r \geq$ 0.99	J / UJ
CCV	Criteria	Action (Detects/Nondetects)
RF	RF \geq 0.05 for all target analytes, including SPCCs	J / R Note: Sample results negated (U) on the basis of blank contamination are not rejected, but estimated (UJ).
%D	%D \leq 20 for CCCs %D \leq 25 for other analytes (20% if no CCCs)	J / UJ low recovery J / Accept high recovery

SPCCs: n-Nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol.

B/N CCCs: Acenaphthene, 1,4-dichlorobenzene, hexachlorobutadiene, diphenylamine, di-n-octylphthalate, fluoranthene, and benzo(a)pyrene

Acid CCCs: 4-Chloro-3-methylphenol, 2,4-dichlorophenol, 2-nitrophenol, phenol, pentachlorophenol, and 2,4,6-trichlorophenol

* A separate worksheet should be filled out for each initial curve.

V. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

1. Frequency Requirements

Was a preparation blank analyzed for each matrix, at the frequency stated in the method? Yes or No

If "No", data quality may be affected. Use professional judgment to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

2. Blanks: List the contamination in all of the blanks (laboratory and/or field QC blanks) below. High and low level blanks must be treated separately.

[illegible]

3. Blank Actions

The action level (AL) for samples which have been diluted should be multiplied by the dilution factor.

No detected sample result should be reported unless the concentration of the compound in the sample exceeds the AL of 10x the amount in the blank for the common contaminants (phthalates), or 5x the amount for any other compound. Specific actions are as follows:

1. If sample result is \leq the sample quantitation limit (SQL) and \leq the AL, report the compound as not detected (U) at the SQL.
2. If sample result is $>$ SQL but \leq AL, report the compound as undetected (U) at the reported concentration.
3. If the concentration is $>$ the AL, report the concentration unqualified.

VI. SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The effectiveness of the analysis is measured by the surrogate percent recovery (%R). Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgement.

List the %Rs which do not meet the criteria for surrogate recovery.

Sample ID	Surrogate Compounds				Matrix: Aqueous/Soil		
	NBZ	FBP	TPH	PHL	2FP	TBP	Actions
QC limits (LL - UL) must be filled in during validation.							
Aqueous	to	to	to	to	to	to	
Solid	to	to	to	to	to	to	

Notes:

Base / Neutral Surrogates

NBZ = Nitrobenzene-d5

FBP = 2-Fluorobiphenyl

TPH = Terphenyl-d14

Acid Surrogates

PHL = Phenol-d5

2FP = 2-Fluorophenol

TBP = 2,4,6-Tribromophenol

Criteria:

- Surrogate recoveries must fall between the QC limits established for the project. If any surrogate is out of QC limits, reanalysis is recommended to confirm that the noncompliance is due to sample matrix effects rather than laboratory deficiencies.

Actions: Data are not qualified unless

- Two or more surrogate %Rs within the same fraction (base/neutral or acid) are out of specification but >10% or
- One surrogate %R within the same fraction <10%.

Surrogate action should be applied as follows:

Qualify results within the same fraction (base/neutral or acid)	%R		
	< 10%	10%-LL	> UL
Detected Results	J	J	J
Non-detected Results	R	UJ	Accept

Note: Sample results negated (U) on the basis of blank contamination are not rejected, but estimated (UJ).

DATA REVIEW WORKSHEETS

All criteria were met _____
 Criteria were not met
 and/or see below _____

VII. MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long-term precision and accuracy of the analytical method for various matrices.

1. MS/MSD Recoveries and Precision (A separate worksheet should be used for each MS/MSD pair)

Sample ID: _____ Level / Matrix: _____

List the %Rs and relative percent differences (RPDs) of compounds that do not meet the project QC criteria.

Note: RPDs are calculated from MS and MSD concentrations, not recoveries.

MS or MSD	Compound	%R or RPD	QC Limits	Action
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

No action is taken on MS/MSD results **alone** to qualify an entire case. However, the reviewer may use MS/MSD results in conjunction with other QC criteria and determine the need for qualification of the data. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, then qualification should be limited to this sample alone. However, it may be determined through MS/MSD results that the laboratory is having a systematic problem in the analysis of one or more analytes (which affects all associated samples), then qualification should be applied to all samples in the analytical batch.

Actions: Qualify the unspiked sample as follows:

Qualify results	MS, MSD %Rs			MS/MSD RPD > QC Limit
	<10%	10%-LL	>UL	
Detected Results	J	J	J	J
Non-Detected Results	R	UJ	Accept	UJ

Note: Sample results negated (U) on the basis of blank contamination are not rejected, but estimated (UJ).

2. MS/MSD - Unspiked Compounds

List the concentrations of the unspiked compounds and determine %RSDs in the sample, MS and MSD.

Compound	Concentration			%RSD	Action
	Sample	MS	MSD		
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

Criteria: None specified, use % RSD \leq 50 as professional judgment.

Action:

- If the %RSD > 50, qualify the result in the unspiked sample as estimated (J).
- If the %RSD is not calculable (NC) due to a nondetect value in the sample, MS, and/or MSD, use professional

DATA REVIEW WORKSHEETS

All criteria were met _____

Criteria were not met
and/or see below _____

judgment to qualify sample data.

VIII. LABORATORY CONTROL SAMPLE (LCS) or LCS/LCS DUPLICATE

This data is generated to determine accuracy of the analytical method for various matrices.

1. LCS or LCS/LCS Duplicate: List the %Rs and/or RPDs of compounds which do not meet the criteria.

[illegible]

Criteria:

- Project QC limits (LL = lower limit, UL = upper limit)

Actions: Actions on LCS %R and RPD should be based on both the number of compounds that are outside the %R criteria and the magnitude of the exceedance of the criteria.

	LCS, LCSD %R			LCS/LCSD
Qualify results	<10%	10%-LL	>UL	RPD > QC Limit
Detected Results	J	J	J	J
Non-Detected Results	R	Use professional judgement	Accept	UJ

Note: Sample results negated (U) on the basis of blank contamination are not rejected, but estimated (UJ).

- If \leq half of LCS/LCSD compounds are outside the QC limits, qualification applies **ONLY** to the affected analytes.
- If more than half of LCS/LCSD compounds are outside the QC limits, qualification applies to **ALL** affected analytes.

2. LCS Frequency

Was an LCS analyzed at the proper frequency (1 per batch of 20 samples or less per matrix) ?

If “no”, note in validation memo and use professional judgment in qualification of the data. Discuss any actions below:

Sample IDs: Matrix:

[illegible]

Criteria (in the absence of project-specific criteria):

- Actions:**

- If the RPD criterion is exceeded, estimate detected results (J) in the sample and duplicate.

If the sample and/or duplicate are NDs, the RPD is not calculable:

- If both the sample and duplicate results are ND, precision is considered acceptable and no action is needed.
- If one sample result is ND and the other is $\geq 5 \times \text{SQL}$, qualify both results (J/UJ).
- If one sample result is ND and the other is $< 5 \times \text{SQL}$, accept unqualified.

Note: If the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is required for CCVs and recommended for samples to assist the data reviewer in determining the condition of the analytical instrumentation or effect of matrix on sample results.

List the IS area and/or retention times (RTs) which do not meet the criteria for IS performance.

[illegible]

Criteria:

- IS area of the CCV must fall between -50 and 100% of the IS area of the midpoint in the ICAL.
- IS RT of the CCV must fall between ± 30 second of the IS RT of the midpoint of the ICAL.
- IS area of the sample must fall between -50% to +100% of the IS area in the associated CCAL.
- IS RT of the sample must fall between ± 30 seconds of the IS RT in the CCAL.

Actions:

If an IS is out of QC limits, reanalysis is recommended to confirm that the noncompliance is due to sample matrix effects rather than laboratory deficiencies.

1. Validation action should be applied to the compounds quantitated with the out-of control IS as follows:

Qualify results	Sample IS area compared to CCAL		
	Extreme low (< -10%)	-10% to -50%	> +100%
Detected Results	J	J	J
Non-Detected Results	R	UJ	Accept

2. 2.If RT of an IS varies more than 30 seconds from the CCV, reject (R) all nondetects in the affected samples.

Discuss any actions below:

The compound identification evaluation is to verify that the laboratory correctly identified target analytes as well as the tentatively identified compounds (TICs).

1. Verify that the target analytes were within the retention time windows and spectra match.
2. Verify that target analytes and/or TICs were quantitated using the correct internal standards.
3. Verify that the target identification is supported by the mass spectral pattern.

I. QUANTITATION LIMITS and SAMPLE RESULTS

The sample quantitation evaluation is to verify laboratory quantitation results.

1. In the space below, please show a minimum of one sample calculation.

2. If dilutions performed, were the SQLs elevated accordingly by the laboratory? List the affected samples and dilution factor in the table below.

Sample ID	Dilution Factor	Reason for Dilution

If dilution was required but not performed, estimate results (J) for the affected compound.

List the affected samples/compounds: _____

3. If requested for the project, verify that results below the SQL and above the laboratory's method detection limits (MDLs) were reported.

4. Verify that the reporting limit is at or above the lowest calibration standard.

5. Verify that results were reported in dry weight for solid samples.

6. Are all sample percent solid ≥ 30 ? If not, list the affected samples/percent solids.

DATA REVIEW WORKSHEETS

Type of Review Full: _____
 Limited: _____ENSR Data Pkg ID _____
Site Name: _____
Project Number: _____

REVIEW OF RADIOLOGICAL DATA PACKAGE

The following guidelines for evaluating radiological data were created to delineate required review actions. This document will assist the reviewer in using professional judgement to make more informed decisions and in better serving the needs of the data users. The radiological data will be reviewed based on method compliance and quality control (QC) results to provide a level of assurance that an nuclide is present or absent. The level of uncertainty (bias) associated with the reported result will be indicated, if applicable. The evaluation of the radiological data will be evaluated based on the Department of Energy *Evaluation of Radiochemical Data Usability* (1997). However, the QC samples will not be evaluated based on a statistical level of confidence as discussed in the *Evaluation of Radiochemical Data Usability* (1997), but rather to the laboratory QC acceptance criteria, unless otherwise indicated.

The hardcopied (laboratory name) _____ data package received has been reviewed and the quality control (QC) and performance data summarized. The review of radiological data included:

Lab Project/SDG No.: _____
Sample Matrix: _____
No. of Samples: _____
Field Blank ID: _____
Equipment Blank ID.: _____
Field Duplicate IDs.: _____List analyses reviewed and analytical method: _____

The general criteria used to determine performance were based on an examination of (check all that apply):

<input type="checkbox"/> Data Completeness	<input type="checkbox"/> Matrix Spike/Matrix Spike Duplicate (MS/MSD)
<input type="checkbox"/> Holding Times/Sample Preservation	<input type="checkbox"/> Laboratory Duplicates
<input type="checkbox"/> Method Blank	<input type="checkbox"/> Field Duplicates
<input type="checkbox"/> Chemical Yield (Tracers and Carriers)	<input type="checkbox"/> Sample Identification and Quantitation
<input type="checkbox"/> Laboratory Control Sample (LCS)	<input type="checkbox"/> Reporting Limits
<input type="checkbox"/> Calibration	

Overall Comments: _____

Definitions and Qualifiers:

- U - Nuclide considered not detected above the reported Minimum Detectable Concentration (MDC) or 2 sigma counting uncertainty
- J - Nuclide identified; the associated numerical value is estimated
- UJ - Nuclide is not detected above the reported MDC or 2 sigma counting uncertainty; the reporting limit may be inaccurate or imprecise
- R - Result is rejected and is not usable for project objectives

In general, only one qualifier is permitted with each result. Qualifiers relating to identification (U or R) take precedence over qualifiers relating to quantitation (J or UJ). Whenever an "R" is used for nondetects, "UJ" is not used. Within each category of qualifiers, use the qualifier that indicates a more serious problem.

Reviewer: _____ Date: _____

DATA REVIEW WORKSHEETS

All criteria were met: _____
Criteria were not met,
and/or see below: _____

I. DATA COMPLETENESS**A. Data Package:**

- _____ The tests requested on the COC or in subsequent communications were performed and reported
- _____ The correct nuclide list was reported
- _____ The COCs (external and internal) are present and properly completed
- _____ Sample receiving documentation is complete

Missing InformationDate Lab ContactedDate Received

B. Other Discrepancies

Codes

SR – Sample Results

BR – Blank Result

MDC – Minimum Detectable Concentration

TPU – Total Propagated Uncertainty

RL- Reporting Limit

DATA REVIEW WORKSHEETS

All criteria were met: _____
 Criteria were not met,
 and/or see below: _____

II. HOLDING TIMES (continued)

The objective of this parameter is to ascertain the validity of results based on the holding time of the sample from the time of collection to the time of sample analysis (activity detection). Samples must be analyzed prior to significant decay of short-lived target radionuclides. Complete the table for all samples and circle the analysis date for samples not within criteria.

SAMPLE ID	DATE SAMPLED	Ra-226 DATE ANALYSIS	Ra-228 DATE ANALYSIS	Tc-99 DATE ANALYSIS	Tritium DATE ANALYSIS	pH	ACTION

Cooler Temperature: _____

Criteria:

- Analysis Holding Times: no technical holding times due to long half lives, but 6 months from sample collection (for contractual reasons)
- Sample Preservation: Concentrated HCL or HNO₃ to pH ≤ 2 for gross alpha or beta, Ra-226, Ra-228, isotopic uranium, isotopic thorium, and Tc-99

Actions:

If samples not preserved properly in the field or laboratory and/or stored in improper container, then:

- SR < sample MDC qualify as estimated "UJ".
- SR ≥ sample MDC use professional judgment to qualify as estimated "J"

DATA REVIEW WORKSHEETS

All criteria were met: _____
Criteria were not met,
and/or see below: _____

III. BLANK ANALYSIS RESULTS

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply to any blank associated with the samples, including equipment, field, and laboratory blanks.

If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

1. Frequency Requirements

Was a method blank analyzed at the frequency stated in the method or by the project? Yes or No
Was the method blank the same matrix as the sample in the batch? Yes or No

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify the data accordingly. Discuss any actions below, and list the samples affected.

2. Blank Actions

The method blank activities must be less than their MDC and 2 sigma counting uncertainty.

Blanks must be evaluated in the following order:

- Method blanks must first be used to qualify equipment/field blanks and samples.
- Contamination remaining in the equipment/field blanks will be used to qualify the associated samples.

Actions:

- if blank results < MDC or < 2 sigma counting uncertainty – no action
- if blank results > MDC , but SR < sample MDC – no action
- if blank results > MDC and SR > sample MDC or 2 sigma counting uncertainty, then
 - determine normalized absolute difference between blank and SR using

$$\frac{\text{Absolute Difference (SR – BR)}}{\text{Square Root (TPU}_{\text{SR}}^2 + \text{TPU}_{\text{BR}}^2)}$$

If normalized absolute difference > 2.58, no qualification

If normalized absolute difference between 1.96 and 2.58, qualify SR \geq MDC as estimated “J”

If normalized absolute difference between 0 and 1.96, use professional judgment to “R”

DATA REVIEW WORKSHEETS

All criteria were met: _____
Criteria were not met,
and/or see below: _____

III. BLANK ANALYSIS RESULTS (continued)

List the contamination > MDC or RL in Sections A & B below.

A. Method blanks

Matrix: _____ Unit _____

[illegible]

B. Field/Equipment blanks

Date Collected	Field ID	Nuclide	Concentration	RL	Affected Samples

C. Normalized Absolute Difference

[illegible]

DATA REVIEW WORKSHEETS

All criteria were met: _____
 Criteria were not met,
 and/or see below: _____

IV. CHEMICAL YIELD (TRACERS AND CARRIERS)

Tracers and carriers used in radiochemical separation methods are used to evaluate chemical separation.

1. Frequency Requirements

Were carrier or tracer percent recoveries reported for each sample? Yes or No

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

2. Carrier or Tracer Recovery

List samples that have carrier or tracer percent recoveries (%Rs) outside criteria.

Sample ID	Nuclide	%R	Action

Criteria: %R = 25-125% for isotopic uranium

Actions: Do not qualify data on yield results alone. If carrier or tracer %Rs are low, there may be increased uncertainty in the SR (MDC > RL). If the yield is low, but the LCS %Rs are acceptable, then accept data without qualification.

DATA REVIEW WORKSHEETS

All criteria were met: _____
 Criteria were not met,
 and/or see below: _____

V. LABORATORY CONTROL SAMPLE (LCS)

The assessment of the LCS(s) is to monitor the accuracy of preparation and analysis.

1. Recovery Criteria

List LCS percent recoveries (%Rs) or normalized differences not within the criteria and the samples affected.

Date	Nuclide	%R/Normalized Difference	Action	Sample Affected
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

Criteria: _____ %R = 75-125% or _____ list other %R or _____ Normalized Difference

Actions:

If %R criteria used then follow the actions stated below:

LCS	%R < 10%	%R = 10 – LL%	%R > UL%
Detected Sample Results	R	J	J
Nondetected Results	R	UJ	Accept

LL – lower limit

UL – upper limit

If normalized difference criteria used then follow the actions stated below:

LCS	Negative bias less than -2.58	Negative bias between -1.96 and -2.58	Between -1.96 and 1.96	Positive bias between 1.96 and 2.58	Positive bias greater than 2.58
Sample Results > MDC	R*	J	Accept	J	R*
Sample Results < MDC	R*	UJ	Accept	Accept	Accept

* Consider the effects of other QC samples prior to qualifying

2. Frequency Criteria

Was an LCS analyzed with each batch?

Yes or No

Was the LCS analyzed on the same detection system as the samples?

Yes or No

If no, data quality may be jeopardized. Use professional judgment to determine the severity of the effect and qualify the data accordingly. Discuss any actions and list affected samples.

DATA REVIEW WORKSHEETS

All criteria were met: _____
 Criteria were not met,
 and/or see below: _____

VI. MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)
1. Recovery Criteria

Sample # _____ Matrix: _____ Units: _____

This data is generated to determine long-term precision and accuracy of the analytical method for various matrices.

List MS/MSD percent recoveries (%Rs) or normalized differences not within the criteria and the samples affected.

Nuclide	Spiked Sample Result (SSR)	Sample Result (SR)	Spike Added (S)	%R	%RPD	Normalized Difference	Action

Criteria: _____ %R = 75-125% or _____ list other %R or _____ Normalized Difference

Actions: MS/MSD actions apply to the field sample used for the MS/MSD analyses. This qualification may also be applied to the results of all samples within a given area of the site or preparation batch, if deemed appropriate.

If %R criteria used then follow the actions stated below:

Qualify Results	MS and/or MSD %R			-20% < %RPD > 20%
	%R < 10%	%R = 10 - LL%	%R > UL%	
Detected Results	J	J	J	J
Nondetected Results	R	UJ	Accept	UJ

LL – lower limit

UL – upper limit

If normalized difference criteria used then follow the actions stated below:

LCS	Negative bias less than -2.58	Negative bias between -1.96 and -2.58	Between -1.96 and 1.96	Positive bias between 1.96 and 2.58	Positive bias greater than 2.58
Sample Results > MDC	R*	J	Accept	J	R*
Sample Results < MDC	R*	UJ	Accept	Accept	Accept

* Consider the effects of other QC samples prior to qualifying

2. Frequency Criteria

Was a matrix spike prepared at the frequency stated in the method or by the project? Yes or No
 Were all nuclides or interest spiked into the MS/MSD? Yes or No or NA

DATA REVIEW WORKSHEETS

All criteria were met: _____
Criteria were not met,
and/or see below:

VII. LABORATORY DUPLICATES*

Sample # _____ Matrix: _____ Units: _____

Laboratories run duplicate samples to verify laboratory consistency and precision. They are a measure of laboratory performance. It is expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with preparing identical duplicate samples.

List nuclide not meeting the RPD or Normalized Absolute Difference (circle criteria used).

[illegible]

Laboratory duplicate actions should be applied to the field sample used as the laboratory duplicate. This qualification may also be applied to the results of all samples within a given area of the site and/or preparation batch, if deemed appropriate.

Criteria:

- RPD $\pm 20\%$ for aqueous, RPD $\pm 35\%$ for soil samples, if sample and duplicate results $\geq 5 \times$ RL or MDC.
- QC limits \pm RL or MDC for aqueous, $\pm 2 \times$ RL or MDC for soil samples, if sample/duplicate results $< 5 \times$ RL or MDC.
- Normalized absolute difference less than or equal to 1.96

Actions: Indicate which criteria were used to evaluate precision by circling RPD, RL, or MDC. If both samples are nondetected, precision is considered acceptable. No action is needed.

- If RPD is exceeded and sample results are $\geq 5x$ RL or MDC, estimate detected results and nondetects (J/UJ).
- RPD is exceeded and sample or duplicate result is $< 5x$ RL or MDC, estimate detected results and nondetects (J/UJ) for nuclides whose absolute difference is $> RL$ or MDC for waters or $> 2x$ RL or MDC for soils.
- If normalized absolute difference is greater than 1.96, estimate results (J/UJ)

2. Frequency Criteria

Was a laboratory duplicate prepared and analyzed with each batch of up to 20 samples? Yes or No

*A separate worksheet page should be used for each laboratory duplicate

DATA REVIEW WORKSHEETS

All criteria were met: _____
 Criteria were not met,
 and/or see below: _____

VIII. FIELD DUPLICATES*

Sample # _____ Matrix: _____ Units: _____

Field duplicate samples may be taken and analyzed as an indication of overall precision. Field duplicate analyses measure both field and lab precision; therefore, the results may have more variability than lab duplicates which measure only lab performance. It is also expected that solid matrices will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

List nuclide not meeting the RPD or Normalized Absolute Difference (circle criteria used).

Nuclide	MDC	RL	Sample Results	Field Duplicate Results	RPD (%)	Normalized Absolute Difference	Action

Field duplicate actions should be applied to the field duplicate pair. This qualification may be applied to the results of all samples within a given area of the site and/or preparation batch, if deemed appropriate.

Criteria:

- RPD $\pm 30\%$ for aqueous, $\pm 50\%$ for soils, if sample and duplicate results $\geq 10x$ RL or MDC.
- Absolute difference $\pm 4x$ RL or MDC for aqueous, $\pm 8x$ RL or MDC for soils, if sample and duplicate $< 10x$ RL or MDC.
- RPD and absolute difference must be exceeded if one result $\geq 10x$ RL and one $< 10x$ RL.
- Normalized absolute difference less than or equal to 1.96

Actions: Indicate which criteria were used to evaluate precision by circling RPD, RL, or MDC. If both samples are nondetected, precision is considered acceptable. No action is needed.

- If RPD is exceeded and sample results are $\geq 10x$ RL or MDC, estimate detected results and nondetects (J/UJ).
- If RPD is exceeded and sample or duplicate result is $< 10x$ RL or MDC, estimate detected results and nondetects (J/UJ) for elements whose absolute difference is $> 4x$ RL or MDC for waters or $> 8x$ RL or MDC for soils.
- If RPD is NC because one result $\geq 10x$ RL or MDC and one nondetect, estimate detected and nondetects (J/UJ).
- If RPD is NC because one result $< 10x$ RL or MDC and one nondetect, use professional judgment. Laboratory duplicate actions should be applied to the field sample used as the laboratory duplicate.
- If normalized absolute difference is greater than 1.96, estimate results (J/UJ)

2. Frequency Criteria

Was a laboratory duplicate prepared and analyzed with each batch of up to 20 samples? Yes or No

*A separate worksheet page should be used for each laboratory duplicate

DATA REVIEW WORKSHEETS

All criteria were met: _____
 Criteria were not met,
 and/or see below: _____

IX. CALIBRATION

1. Standard Traceability

- a. Were certificates included for calibration standards, LCS, and/or MS/MSD? Yes or No
- b. Did certificate serial numbers match referenced standards? Yes or No
- c. Were the standards within the expiration dates? Yes or No

If no, list standards affected

Standard ID	Nuclide	Lab ID	Certificate ID	Expiration Date

2. Calibration Verification

- a. Are the efficiencies within the appropriate control criteria? Yes or No
- b. Are instrument backgrounds within the appropriate control criteria? Yes or No
- c. Are energies within the appropriate control criteria? Yes or No
- d. Peak resolution within appropriate control criteria? Yes or No

If no, list standards affected

Standard ID	Nuclide	Lab ID	Certificate ID	Expiration Date

DATA REVIEW WORKSHEETS

All criteria were met: _____
 Criteria were not met,
 and/or see below: _____

X. SPECTRAL INTERPRETATION

1. Gamma Analyses

- a. Do isotopes of the same radionuclide show secular equilibrium? Yes or No
- b. Soil samples: are peaks at 511 keV (annihilation peak) and 1460 keV (K-40) present? Yes or No
- c. Are all detected peaks correctly identified? Yes or No
- d. Do peaks overlap? Yes or No

If yes, list affected samples and nuclides

Sample ID	Nuclide	Peak Energy	Estimated % Overlap	Action

2. Alpha Spectra

- a. Are target peaks within the energy range of interest (ROI)? Yes or No
 - b. Does peak overlap exist through tailing from other nuclides? Yes or No
- If yes, list samples and nuclides affected by tailing below:

Sample ID	Nuclide	Issue

DATA REVIEW WORKSHEETS

All criteria were met: _____
 Criteria were not met,
 and/or see below: _____

XI. SAMPLE IDENTIFICATION AND QUANTITATION

1. Are sample results > sample MDCs? Yes or No
 If no, qualify SRs as "U". List samples and nuclides below

Sample ID	Nuclide	SR	SR MDC	Action

2. Are sample results > 2 sigma counting uncertainty? Yes or No
 If no, qualify SRs as "U". List samples and nuclides below

Sample ID	Nuclide	SR	SR MDC	Action

Use Professional judgment in cases where:

- SR < MDC, but > 2 sigma counting uncertainty may have been counted long enough to be considered detected.
- SR > MDC, but < 2 sigma counting uncertainty may NOT have been counted long enough to be considered detected.

3. Are sample results > 2 TPU? Yes or No
 If no, qualify sample results (SRs) as "U". List samples and nuclides below

Sample ID	Nuclide	SR	SR TPU	Action

Use Professional judgment in cases where:

- SR < MDC, but > 2 sigma counting uncertainty may have been counted long enough to be considered detected.
- SR > MDC, but < 2 sigma counting uncertainty may NOT have been counted long enough to be considered detected.

DATA REVIEW WORKSHEETS

All criteria were met: _____
 Criteria were not met,
 and/or see below: _____

4. Negative Sample Results

Negative results with absolute values greater than their 2 sigma counting uncertainty indicate that the instrument background may have shifted. Use professional judgment to qualify data.

Do negative sample results have absolute values > 2 sigma counting uncertainty? Yes or No
 If yes, list samples and nuclides below:

Sample ID	Nuclide	SR	SR 2 sigma uncertainty	Action

5. Gross values vs total of individual nuclides

Are gross alpha results > total of individual uranium results Yes or No
 If no, list samples with gross alpha < total of individual uranium isotopes

Sample ID	Gross alpha SR	Total of individual uranium isotopes	Action

Action

- if gross alpha < total of individual isotopic uranium, then estimate (J) detected individual U results if applicable in affected sample.

DATA REVIEW WORKSHEETS

All criteria were met: _____
 Criteria were not met,
 and/or see below: _____

XII. REPORTING LIMITS

1. Minimum Detectable Concentration (MDC)

A. Were sample MDCs < RLs?

Yes or No

B. Determine why the MDC > RL (ex. small sample size, inadequate count time, or matrix problems). If sample MDC > RL and SR < sample MDC or ± 2 sigma counting uncertainty, and there is no justification for not reanalyzing at a longer count time or greater sample aliquot, then data are noncompliant with RL – note in report. The data may be affected. Use professional judgment to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

2. Aliquot Size

List samples and nuclides that required adjusted aliquot size.

Sample ID	Nuclide	Aliquot Size

A representative sample aliquot must be chosen to ensure the dissolved solid content of the sample falls within the mass range of the appropriate curve. Sample results for which aliquot weight is outside the attenuation curve should be qualified as estimated (J) if not reanalyzed with a smaller aliquot.

DATA REVIEW WORKSHEETS

Type of Validation Full: _____
Limited: _____

ENSR Data Pkg#: _____

Site Name: _____

Project Number: _____

REVIEW OF METALS ANALYSIS DATA PACKAGE

The following guidelines for evaluating metals were created to delineate required validation actions. This document will assist the reviewer in using professional judgement to make more informed decisions and in better serving the needs of the data users. Quality control validation criteria were derived from USEPA publications: "Test Methods for Evaluating Solid Waste, Physical / Chemical Methods SW846" (3RD Edition and subsequent Updates), specifically SW-846 methods 6010B, 7470A, and 7471A. Validation actions were derived from "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review" (Final, October 2004) and were modified to accommodate the non-CLP methods and professional judgment. The project QAPP should be reviewed for project-specific information.

The hardcopy data package received from (laboratory name/location) _____ has been reviewed and the quality assurance and performance data summarized.

The data review for metals included:

Lab Project/SDG No. _____ No. of Samples: _____ Sample Matrix: _____
Field Blank IDs: _____
Equipment Blank IDs.: _____
Field Duplicate IDs.: _____

The general criteria used to determine the performance were based on an examination of (check all that apply):

_____ Data Completeness	_____ Laboratory Duplicate Results
_____ Holding Times and Sample Preservation	_____ Field Duplicate Results
_____ Calibrations	_____ Laboratory Control Sample
_____ Blanks	_____ ICP Serial Dilution Results
_____ ICP Interference Check Sample	_____ Sample Quantitation Assessment
_____ Matrix Spike Results	_____ GFAA Results (Addendum)

NOTE: If GFAA methods (SW-846 7000 series) were used to analyze samples, the data validation of these analyses should be attached as an addendum to these Data Review Worksheets.

If spreadsheets are used to automate calculations, they must be attached to these worksheets.

Overall Comments: _____

Definitions and Qualifiers:

J - Estimated result with undetermined bias
J+ - Estimated result, result may be biased high

U - analyte not detected
UJ - Estimated nondetect – quantitation limit is estimated

J- - Estimated result, result may be biased low

R - Rejected data (unusable)

QL – quantitation limit (the laboratory may use reporting limit, practical quantitation limit, detection limit)

MDL or IDL – Method detection limit or instrument detection limit, respectively

Actions based on MS, laboratory duplicate, field duplicate, serial dilution analyses should be applied to all samples of the same matrix or to the results of all samples within a given area of the site, if deemed appropriate. Any validation action based upon professional judgement or any deviation from the validation guidelines presented in these worksheets needs to be described in detail in an attached narrative or memo.

Reviewer: _____ Date: _____

All criteria were met: _____
Criteria were not met,
and/or see below: _____

I. DATA COMPLETENESS

A. Data Package:

_____ The tests requested on the COC or in subsequent communications were performed and reported.

_____ The correct analyte list was reported.

_____ The COCs (external and internal) are present and complete.

_____ Sample receiving documentation is complete.

Missing Information

Date Lab ContactedDate Received

B. Other Discrepancies

III. INSTRUMENT CALIBRATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrumentation is capable of producing and maintaining acceptable quantitative data.

1. Analytical Sequence

- | | |
|---|-----------|
| A. Did the laboratory use the proper number of standards for calibration as described in the method or per manufacturer's recommendation?
(ICP metals = a blank and at least one standard, Hg = a blank and at least five standards) | Yes or No |
| B. Were initial calibrations performed successfully on a daily basis or once/24 hours and each time the instrument was set up? | Yes or No |
| C. Were all measurements the average of at least two replicate exposures? | Yes or No |
| D. Was an initial calibration verification standard (ICV) analyzed for all analytes at the proper concentration (e.g., within the linear range for 6010B) and at the beginning of sample analysis? | Yes or No |
| Was the ICV made from a second source (different than calibration standards)? | Yes or No |
| E. Were continuing calibration verification standards (CCVs) analyzed for all analytes immediately following daily calibration, every 10 samples, and at the end of the run at a mid-range concentration? | Yes or No |
| F. Although not required by SW-846, if a Contract Required Quantitation Limit (CRQL) check standard was analyzed, was the concentration of this CRQL standard at or comparable to the QL reported by the laboratory?
Note: for ICP analysis, the CRQL check standard is often referred to as CRI standard. For CVAA, the CRQL check standard is often referred to as CRA | Yes or No |

Actions:

1. If the calibration was not performed, qualify all results as unusable (R).
2. If the calibration is incomplete or if any of the above answers are "No", data quality may be affected. Use professional judgment to determine the severity of the effect. Discuss any actions below and list the samples affected.

IV. BLANK ANALYSIS RESULTS

The objective of blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory or field activities. The criteria for evaluation of blanks apply to any blank associated with the samples, (e.g., equipment blank [EB], field blank [FB], preparation blank [PB], initial and continuing calibration blanks [ICB/CCB], etc.). If problems with any blank exist, all data associated with the case must be carefully evaluated to determine if there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting samples.

- | | |
|---|-----------|
| A. Was a PB analyzed for each matrix or with each batch of samples digested (≤ 20 samples)? | Yes or No |
| B. Was an ICB analyzed after the calibration standards ? | Yes or No |
| C. Was a CCB analyzed after every ten samples and at the end of the run? | Yes or No |

Data quality may be affected if any of the above answers are "No". Use professional judgment to determine if the associated sample data should be qualified. Discuss any actions on a separate attached sheet, and list the samples affected.

Actions

Blanks must be evaluated in the following order:

- Lab blanks (preparation and calibration) must first be used to qualify equipment/field blanks and samples.
- Any contamination remaining in the equipment/field blanks will be used to qualify the associated samples.

Full Validation:

1. For PB nonconformance, apply action to all samples in the analytical batch.
2. For ICB nonconformance, apply action to all samples in the analytical sequence.
3. For CCB nonconformance, apply action to samples analyzed between the previous in-control CCB and the subsequent in-control CCB.

Limited Validation: Use the highest blank PB, ICB, or CCB in the analytical batch or sequence.

The blank actions/qualifications on the following page are written for CLP methods and not SW-846. Therefore, professional judgment may be used to modify some of the actions (e.g., in the case where QLs are extremely high or not technically supported). It may be appropriate to use actions for blanks from the 1994 National Functional Guidelines (e.g., if the laboratory reports nondetects at the MDL/IDL). Justification for using this approach must be documented in the worksheets and in the validation memorandum.

The guidelines below should be followed when using the 1994 National Functional Guidelines and the "5x rule".

Establish an Action Level (AL) for any analyte equal to five times (5x) the highest concentration of that element's contamination in any blank. Any blank with a negative result whose absolute value $>$ IDL or MDL (or lowest quantitation limit) must be carefully evaluated to determine its effect on the sample data. Use professional judgment to assess the data.

Blanks must be evaluated in the following order:

- Lab blanks (preparation and calibration) must first be used to qualify equip/field blanks and samples.
- Any contamination remaining in the equip/field blanks will be used to qualify the associated samples.

Actions:

1. For positive blank contamination:
 - results \leq AL are qualified as undetected (U) at the reported concentration.
 - results $>$ AL or nondetects are accepted unqualified.
2. For negative blank contamination:
 - results \leq absolute value of negative AL are estimated (J).
 - nondetects are estimated (UJ).
 - results $>$ AL are accepted unqualified.
3. When both positive and negative blank contaminations exist, use professional judgment to assess data.

October 2004 National Functional Guidelines

Blank Actions

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB (Positive)	\geq MDL but \leq QL	Nondetect	No action
		\geq MDL but \leq QL	Qualify as nondetect (U) at the QL
		> QL	Use professional judgement (see below [1])
	>QL	\geq MDL but \leq QL	Qualify as nondetect (U) at the QL
		> QL but < Blank Result	Qualify as nondetect (U) at the blank level Or qualify result as unusable (R).
		> Blank Result	Use professional judgement (see below [1])
ICB/CCB (Negative)	\leq (-MDL) but \geq (-QL)	\geq MDL or nondetect	Use professional judgement (see below [2])
	< (-QL)	< 10x QL	Qualify results \geq QL as estimated low (J-) and nondetects as estimated (UJ)
		> 10x QL (professional judgment)	No action (professional judgment)
PB / EB / FB (Positive)	> QL	\geq MDL but \leq QL	Qualify as nondetect (U) at the QL
		> QL but < 10x Blank Result	Qualify results as unusable (R) or estimated high (J+)
		\geq 10x Blank Result	No action
	\geq MDL but \leq QL	Nondetect	No action
		\geq MDL but \leq QL	Qualify as nondetect (U) at the QL
PB (Negative)	< (-QL)	> QL	Use professional judgement (see below [1])
		< 10x QL	Qualify results \geq QL as estimated low (J-), non- detects as estimated (UJ)
		> 10x QL (professional judgment)	No action (professional judgment)

[1] Consider establishing an action level (AL) at 5x the blank contamination. If sample result is <AL, qualify the reported result with a "U".

[2] Consider estimating positive results and nondetects (J-/UJ).

IV. BLANK ANALYSIS RESULTS (continued)

Laboratory blanks

Matrix: Solid / Aqueous

Date Analyzed	Prep/ ICB/CCB	Analyte	Concentration (circle highest)	Units	Actions for Samples	Affected Samples

Field/Equipment blanks

Date Collected	Field ID	Analyte	Concentration	Units	Actions for Samples	Affected Samples

The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. For example, soil sample results will not be in the same units as the ICB, CCB, EB, or FB data. It may be easier to work with the raw data or use the following equation to convert results in µg/L to mg/Kg.

ICB, CCB, EB, or FB concentration in µg/L must be converted to mg/kg in order to compare with sample results.

$$\text{Concentration in } \mu\text{g/L} \times \frac{\text{Volume diluted to (ml)}}{\text{Weight digested (g)}} \times \frac{1\text{L}}{1000\text{ml}} \times \frac{1000\text{ g}}{1\text{kg}} \times \frac{1\text{mg}}{1000\text{ }\mu\text{g}} = \text{wet weight (mg/kg)}$$

For each sample, the concentrations are converted to dry weight using the % solids calculation:

$$\frac{\text{Wet weight conc}}{\% \text{ Solids}} \times 100 = \text{Concentration in dry weight (mg/kg)}$$

V. ICP INTERFERENCE CHECK SAMPLE

The ICP interference check sample (ICS) verifies the analytical instrument's ability to overcome interferences typical of those found in samples and verifies the laboratory's interelement and background correction factors.

1. Frequency Requirements

A. Was the ICS solution analyzed at the beginning of each sample analysis run? Yes or No

If no, the data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

B. Did the laboratory analyze an ICS A solution (not required in 6010B)? Yes or No

2. Recovery Criteria

List any elements in the ICS solution, which did not meet the %R criteria. Also evaluate the ICS A if the laboratory performed this analysis. Use professional judgment for actions or use those listed below.

Date	Analyte	%R	Action	Samples Affected
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

Criteria: %R = $100 \pm 20\%$ the true value or the true value $\pm 2x$ the RL (whichever is greater).

Actions: If any analyte does not meet the %R criteria, follow the actions stated below:

Full Validation: Use %R = $100 \pm 20\%$ and apply action to samples analyzed between the previous in-control ICS and the subsequent in-control ICS (if the ICS was analyzed more frequently than the method requirement) if samples contain interferences at levels comparable to or greater than the levels in the ICS.

Limited Validation: Use %R = $100 \pm 20\%$ and apply actions to all samples in the analytical sequence if samples contain interferences at levels comparable to or greater than the levels in the ICS..

Qualify results	%R of Analyte in the ICS Solution		
	%R < 50%	%R = 50%-79% or < true value - 2x RL	%R > 120% or > true value + 2x RL
Detected Results	J-	J-	J+
Nondetects	R	UJ	A

and/or see below:

V. ICP INTERFERENCE CHECK SAMPLE (continued)

3. ICS A Analysis Results (using ENSR professional judgment and guidance from the NFGs since analysis of the ICS A solution is not required in 6010B)

List the concentration of any elements \geq MDL (or lowest quantitation limit used) in the ICS A solution that should not be present. For soil samples, results might not be in the same units as the ICS solutions; it may be easier to work with the raw data.

List the samples affected by interferences below:

[illegible]

Criteria: No target analytes should be present in the ICS A solution at concentration \geq MDL.

Actions:

1. If an element was detected \geq MDL but should not be present in the ICS A and sample concentrations of the interferents (Al, Ca, Fe, and Mg) are $<$ ICS A; accept results unqualified.
2. If an element was detected \geq MDL but should not be present in the ICS A and sample concentrations of the interferents (Al, Ca, Fe, and Mg) are comparable or higher than those found in the ICS A, qualify detected results for the affected element as estimated biased high (J+) and accept nondetects.
3. If an element was detected as negative interference, (i.e., the absolute value \geq MDL) but should not be present in the ICS A and sample concentrations of the interferents (Al, Ca, Fe, and Mg) are comparable or higher than those found in the ICS A, qualify detected results $< 10\times$ the absolute value of the negative result for the affected element as estimated biased low (J-) and nondetects (UJ).

Note: If the levels of interferences in the samples are comparable to or higher than those found in the samples, it may be appropriate to calculate the estimated interference for the analytes of interest using the following equation. Information on the impact of the calculated interference on the results for the analytes of interest may be included in the validation memorandum.

$$\text{Calculated Estimated Interference} = \frac{\text{Interferent in sample}}{\text{Interferent in ICS A}} \times \text{element concentration in ICS A}$$

VII. MATRIX SPIKE (MS) RESULTS

This data is generated to determine the effect of each sample matrix on sample preparation procedures and the measurement methodology.

1. Frequency Criteria

- A. Was the MS analysis performed on a site-specific sample? Yes or No
If no, results are not evaluated due to potential differences in sample matrix.
- B. Was an MS prepared at the required frequency (1 / batch / 20 samples / matrix)? Yes or No
- C. Was a Post digestion spike (PDS) performed for any analytes that fail MS %R criteria? Yes or No
(recommended for a new or unusual matrix and NA for CVAA)
- D. Was a matrix spike/matrix spike duplicate (MS/MSD) analyzed in place of or in addition to a laboratory duplicate analysis? If yes, refer to Section VIII for calculations of RPDs from MS/MSD results. Yes or No

2. Recovery Criteria

List the %Rs for analytes, which did not meet the criteria.

Sample # _____ Matrix: _____ Units: _____

Analyte	MS/MSD Spiked Sample Result (SSR)	Sample Result (SR)	Spike Added (S)	MS/MSD %R	Action

Criteria: %R = 75-125% or project-specific QC limits (LL – lower limit, UL – upper limit).

Actions: MS actions apply to all samples of the same matrix. This qualification will also be applied to the results of all samples within a given area of the site, if deemed appropriate.

- If the sample result (SR) > 4x the spike concentration (S), no action is taken.
- If any analyte does not meet the %R criteria and a Post Digestion Spike analysis was performed, use professional judgement to assess the results. Refer to the National Functional Guidelines for recommended actions.
- If either the MS or MSD does not meet %R criteria, qualify all associated samples.

Qualify results	MS %R in the Sample		
	%R < 30%	%R = 30%- 74% or 30% to LL	%R > 125% or > UL
Detected results	J-	J-	J+
Nondetects	R	UJ	A

VIII. LABORATORY DUPLICATE RESULTS

Laboratories run duplicate samples to verify laboratory consistency and precision. They are a measure of laboratory performance. It is expected that soil/sediment duplicate results will have a greater variance than water matrices due to difficulties associated with preparing identical duplicate samples.

1. Frequency Criteria

- A. Was the duplicate analysis or MSD analysis performed on a site -specific sample? Yes or No
If no, results are not evaluated due to potential differences in sample matrix.
- B. Was a duplicate or MSD analysis prepared at the required frequency (1 /batch /20 samples /matrix)? Yes or No

2. Precision Criteria: List the RPDs for analytes which did not meet the criteria.

Sample # _____ Matrix: _____ Units: _____

For the soil matrix, calculate the sample quantitation limit (RL based on PQL) in mg/kg using the amount, volume, and % solids data for the sample. In some cases the lab may run an MS/MSD in place of a duplicate. Calculate RPDs from MS/MSD results.

Element	QL (ug/L)	QL (mg/Kg)	Sample or MS Result	Duplicate or MSD Result	RPD (%)	Action
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Mercury						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						
Tin						

Attach a separate sheet for additional metals

Criteria:

RPD \pm 20% for aqueous, RPD \pm 35% for soil samples, if sample and duplicate results \geq 5x QL.

QC limits of \pm QL for aqueous, \pm 2x QL for soil samples, if sample or duplicate result $<$ 5x QL.

Actions: Indicate which criteria were used to evaluate precision by circling either the RPD or QL. If both samples are nondetected, the RPD is not calculated (NC), precision is considered acceptable. No action is needed.

If RPD is exceeded and sample or duplicate results are \geq 5x QL, estimate detected results and nondetects (J/UJ).

If RPD is exceeded and sample or duplicate result is $<$ 5x QL (including nondetects) and absolute difference between sample and duplicate is $>$ QL for waters or $>$ 2x QL for soils, estimate detected results and nondetects (J/UJ).

IX. FIELD DUPLICATE RESULTS

Field duplicate samples may be taken and analyzed as an indication of overall precision. Field duplicate analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which measure only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

If appropriate, list the analyte concentration not meeting RPD criteria. For soil matrix, calculate the sample quantitation limit in mg/kg using the amount, volume, and %solids data for the sample.

Sample# _____ Duplicate# _____ Matrix: _____ Units: _____

Element	QL (ug/L)	QL (mg/Kg)	Sample Result	Duplicate Result	RPD (%)	Action
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Mercury						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						
Tin						

Attach a separate sheet for additional metals

Field duplicate actions should be applied to all other samples of the same matrix type. This qualification will also be applied to the results of all samples within a given area of the site, if deemed appropriate.

Criteria:

RPD \pm 30% for aqueous, \pm 50% for soils, if sample and duplicate results $\geq 10x$ QL.

Absolute difference of $\pm 4x$ QL for aqueous, $\pm 8x$ QL for soils if sample and duplicate $< 10x$ QL.

RPD and absolute difference must be exceeded if one result $\geq 10x$ QL and one $< 10x$ QL.

Actions:

Indicate which criteria were used to evaluate precision by circling either the RPD or QL. If both samples are nondetected, the RPD is not calculated (NC), no action is needed.

If RPD is exceeded and sample results are $\geq 10x$ QL, estimate detected results and nondetects (J/UJ).

If RPD is exceeded and sample or duplicate result is $< 10x$ QL, estimate detected results and nondetects (J/UJ) for elements whose absolute difference is $> 4x$ QL for waters and $> 8x$ QL for soils.

If RPD is NC because one result is $\geq 10x$ QL and one nondetect, estimate detected results and nondetects (J/UJ).

If RPD is NC because one result is $< 10x$ QL and one nondetect, use professional judgement.

X. LABORATORY CONTROL SAMPLE (LCS) RESULTS

The assessment of the LCS(s) is to monitor the overall performance of each step during the analysis, including the sample preparation and determine matrix specific precision and accuracy.

Recovery Criteria: List any LCS results not within project-specific criteria, laboratory established control limits or National Functional Guideline recovery criteria.

Indicate which criteria were used: _____

AQUEOUS LCS

Date	Analyte	%R	Action	Samples Affected

Note: NFGs have no control limits for Ag and Sb; however, include Ag and Sb in professional judgment. Apply actions to all samples in the same preparation batch.

Actions:

Aqueous LCS:	%R < 50%	%R = 50 – lower limit or 80%	%R > upper limit or 120%	%R > 150%
Positive Sample Results	J-	J-	J+	R
Nondetects	R	UJ	A	R

SOLID LCS

Date	Analyte	LCS Conc.	QC Windows	Action	Samples Affected

Criteria: LCS results must be within the QC windows provided by the vendor. In absence of vendor limits use aqueous LCS control limits.

Note: Apply actions to all samples in the same preparation batch.

Actions:

Solid LCS	Less than Lower Acceptance Limit	Greater than Upper Acceptance Limit
Positive Sample Results	J-	J+
Nondetects	UJ	Accept

2. Frequency Criteria

Was an aqueous LCS analyzed with each batch of aqueous samples digested or for every group of aqueous samples in an SDG, whichever is more frequent? Yes or No

Was an solid LCS analyzed with each batch of solid samples digested or for every group of soil/sediment samples in an SDG, whichever is more frequent? Yes or No

If no, data quality may be jeopardized. Use professional judgment to determine the severity of the effect and qualify the data accordingly. Discuss any actions and list affected samples.

XI. SERIAL DILUTION ANALYSIS

The assessment of the serial dilution analysis is to determine the effect of the sample matrix on the accuracy of the results.

Were serial dilutions (1:5 dilutions) performed for each matrix and the results of the diluted sample analysis agreed within 10% difference (%D) of the original undiluted analysis for analyte concentrations >50x the IDL or MDL before dilution? Yes or No

Serial dilutions were not performed for the following target analytes: (optional for Hg) Yes or No

Was the serial dilution analysis performed on a site-specific sample? If no, results are not evaluated due to potential differences in sample matrix. Yes or No

List the %Ds for analytes which did not meet the %D criterion (10%).

Sample #: _____ Matrix: _____ Units: _____

Element	IDL/MDL	50x IDL/MDL	Sample Results	Corrected Serial Dilution Results	%D	Action
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Mercury						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						
Tin						

Attach a separate sheet for additional metals

Actions: Actions apply to all samples of the same matrix. This qualification will also be applied to the results of all samples within a given area of the site, if deemed appropriate.

Estimate detected results and nondetects (J/UJ) for elements with %Ds > 10.

XII. SAMPLE QUANTITATION ASSESSMENT

The objective is to ensure that the reported sample quantitation results are accurate. Evaluate any technical problems not previously addressed, examine the raw data for any anomalies, verify that there were no transcription or reduction errors on one or more samples and that results fall within the linear range for ICP and within the calibration range for CVAA.

1. Instrument Detection Limits/Method Detection Limits (IDL/MDL)/Quantitation Limits (QLs):

A. Were results reported down to IDL/MDL or QL? (Circle one) IDL/MDL or QL

B. Were IDL/MDL or QL results for all elements reported at levels that meet project objectives? Yes or No
If not, indicate affected elements:

C. If appropriate, estimate (J) results between the IDL/MDL and QL (refer to project-specific QAPP). Attach a separate sheet listing the qualified samples and analytes.

2. Reporting requirement:

A. Were sample weights (including dry weights), volumes, and dilutions taken into account when reporting results (positive and nondetects)? If no, the reported results may be inaccurate. Request that the laboratory resubmit the corrected data. Yes or No

B. Did sample results fall within the linear dynamic range for ICP and within the calibration range for CVAA? Yes or No
If no, were dilutions performed? Yes or No
List the affected samples/elements/dilution factor :

If no, and dilution was not performed, estimate results (J).
List the affected samples/elements:

XII. SAMPLE QUANTITATION ASSESSMENT (continued)

Sample Quantitation (full validation only): The sample quantitation evaluation is to verify that there were no transcription or reduction errors and to verify laboratory sample quantitation on one or more samples. In the space below, please show a minimum of one sample calculation.

ICP by 6010B

Mercury by 7470A/7471A

For soil samples, the following equation may be necessary to convert raw data values reported in $\mu\text{g/L}$ to actual sample concentrations (mg/kg):

$$\text{Conc. in } \mu\text{g/L} \times \frac{\text{Volume diluted to (ml)}}{\text{Weight digested (g)}} \times \frac{1\text{L}}{1000\text{ml}} \times \frac{1000\text{ g}}{1\text{kg}} \times \frac{1\text{mg}}{1000\text{ }\mu\text{g}} = \text{concentration in wet weight (mg/kg)}$$

In addition, the concentrations are converted to dry weight using the % solids calculation:

$$\frac{\text{Wet weight conc}}{\% \text{ Solids}} \times 100 = \text{Concentration in dry weight (mg/kg)}$$

Laboratory/Location:		ENSR Data Package #:	
Laboratory SDG/Job No:		Client/Site Name:	
No. of Samples-Matrix:		Project Number:	
Acceptance Criteria: QAPP/Method		Validation Actions:	
Validator:	Date Checked:	Full / Limited Validation (circle one)	

DATA PACKAGE COMPLETENESS CHECKLIST

ITEM	YES	NO	N/A	COMMENTS
Sample results included?				
Detection levels included?				
Field I.D. included?				
Laboratory I.D. included?				
Sample matrix included?				
Sample receipt temperature 2-6°C?				
Sample preservation acceptable?				
Signed COCs included?				
Date of sample collection included?				
Date of sample prep. included?				
Date of analysis included?				
Method reference included?				
QC Documentation included?				
Case Narrative included				
Equipment/Field Blank IDs				
Field Duplicate IDs				

Definitions: IDL – Instrument Detection Limit; MDL – Method Detection Limit; RL – Reporting Limit; SQL = Sample Quantitation Limit; %RSD – Percent Relative Standard Deviation; %D – Percent Difference; %R – Percent Recovery; RPD – Relative Percent Difference; r – correlation coefficient; LCS – Laboratory Control Sample; NFG – National Functional Guidelines

Comments	Review Element	Criteria*	Action
	Preserv.:	See method	Use prof. judgment
	HT:	See method	J-/UJ if exceeded
	Calib. curve Cyanide Other	$r \geq 0.995$ $100 \pm 15\%$ $100 \pm 10\%$	Use prof. judgement J+ if exceeded J-/UJ if below
	Blank	< RL	Refer to NFG
	MS/MSD	%R= 75-125% RPD $\pm 20\%$	J+ if %R > 125 J-/UJ if %R < 75 J if RPD exceeded
	Lab Dup	Aq. RPD $\pm 20\%$ So. RPD $\pm 35\%$	J if RPD exceeded
	Field Dup (ENSR)	Aq. RPD $\pm 30\%$ So. RPD $\pm 50\%$	See ENSR DV actions
	LCS/LCSD	%R= 75-125% RPD $\pm 30\%$	J+ if %R > 125 J-/UJ if %R < 75 J if RPD exceeded
* If no criteria specified by the method, lab, or QAPP, use these QC limits as guidance.			

QA/QC CHECKLIST FOR GENERAL CHEMISTRY ANALYSIS

ITEM	YES	NO	N/A	COMMENTS
PARAMETER:		METHOD:		
Calibration Info Included in Lab Package?				
Criteria met? (%RSD, r, %Rs)				
Method Blank Data Included in Lab Package?				
Criteria met? (< RL)				
Field/Equipment Blank Included in Lab Package?				
Criteria met? (< RL)				
Matrix Spike (MS) Data Included in Lab Package?				
%R criteria met? (Method or Lab or QAPP)				
MS Duplicate or Lab Dup Data Included in Lab Package?				
%R or RPD criteria met? (Method or Lab or QAPP)				
Field Duplicate Included in Lab Package?				
RPD criteria met? (QAPP OR ENSR)				
QC Check Samples/LCS Data Included in Lab Package?				
%R criteria met? (Method or Lab or QAPP)				
PARAMETER:		METHOD:		
Calibration Info Included in Lab Package?				
Criteria met? (%RSD, r, %Rs)				
Method Blank Data Included in Lab Package?				
Criteria met? (< RL)				
Field/Equipment Blank Included in Lab Package?				
Criteria met? (< RL)				
Matrix Spike (MS) Data Included in Lab Package?				
%R criteria met? (Method or Lab or QAPP)				
MS Duplicate or Lab Dup Data Included in Lab Package?				
%R or RPD criteria met? (Method or Lab or QAPP)				
Field Duplicate Included in Lab Package?				
RPD criteria met? (QAPP OR ENSR)				
QC Check Samples/LCS Data Included in Lab Package?				
%R criteria met? (Method or Lab or QAPP)				
PARAMETER:		METHOD:		
Calibration Info Included in Lab Package?				
Criteria met? (%RSD, r, %Rs)				
Method Blank Data Included in Lab Package?				
Criteria met? (< RL)				
Field/Equipment Blank Included in Lab Package?				
Criteria met? (< RL)				
Matrix Spike (MS) Data Included in Lab Package?				
%R criteria met? (Method or Lab or QAPP)				
MS Duplicate or Lab Dup Data Included in Lab Package?				
%R or RPD criteria met? (Method or Lab or QAPP)				
Field Duplicate Included in Lab Package?				
RPD criteria met? (QAPP OR ENSR)				
QC Check Samples/LCS Data Included in Lab Package?				
%R criteria met? (Method or Lab or QAPP)				

Use this space for notes/comments and/or spot check calculation (if required)